PRODUCTION OF HIGH TEMPERATURE GREASE FROM WASTE LUBRICANT SLUDGE AND SILICONE OIL

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ABSTRACT

This research was carried out to study the production of high temperature grease from waste lubricant sludge and silicone oil. The effects of different ratios of waste lubricant sludge to fumed silica and mixing time on grease characteristics were investigated. The ratios of waste lubricant sludge and fumed silica used were 100:0, 90:10, 80:20, 70:30, 60:40, 50:50 and 40:60. In terms of mixing time, it was varied at 1, 2, 3, 4 and 5 hours. Throughout the experiment, the grease was prepared by using the heating and mixing technique. The grease produced was analysed according to the ASTM and NLGI standard via penetration test, dropping point test, copper corrosion test, FTIR and AAS. . Fourier transform infra red (FTIR) analysis was carried out to study on the functional groups present in the grease. Atomic absorption spectroscopy analysis was also carried out to determine the concentration of a specific metal element in the grease. The results showed that the grease produced with a minimum ratio of fumed silica to sludge (10:90) was able to produce high temperature grease with a dropping point of 272.5^{o}C. Further analysis verified that the grease is a high temperature grease, has worked penetration of 270-290 which makes the grease fall into the common grease category, has a low tendency to corrode copper and very little metal element was present in the grease. It can be concluded that the ratio of fumed silica to sludge and the mixing time of the grease produced significant effect on worked penetration and dropping point of the grease.

ABSTRAK

Kajian ini dijalankan dengan tujuan mengkaji penghasilan gris bersuhu tinggi daripada sisa pelumas lumpur dan minyak silikon. Kesan daripada pengunaan nisbah yang berlainan bagi sisa pelumas lumpur kepada silika kesal dan masa campuran ke atas ciri-ciri gris telah dikaji. Nisbah sisa pelumas lumpur dan silka kesal yang digunakan adalah 100:0, 90:10, 80:20, 70:30, 60:40, 50:50 dan 40:60. Masa campuran yang digunakan adalah 1, 2, 3, 4 dan 5 jam. Sepanjang eksperimen dijalankan, gris telah dihasilkan dengan menggunakan teknik memanas dan mencampur. Gris yang dihasilkan telah dianalisi mengikut standard ASTM dan NLGI dengan menggunakan ujian penetrasi, ujian titik jatuhan, ujian penghakisan kuprum, FTIR dan AAS. Analisis infra merah merubah fourier (FTIR) telah dijalanan untuk mengkaji kehadiran kumpulan berfungsi di dalam gris. Spektroskopi penyerapan atom (AAS) dijalankan untuk mengkaji konsentrasi unsur logam spesifik di dalam gris. Keputusan menunjukkan bahawa gris yang dihasilkan dengan nisbah minima silika kesal kepada sisa pelumas lumpur (10:90) dapat menghasilkan gris bersuhu tinggi dengan titk jatuhan pada 272.5^{o}C. Analisis lanjut memastikan bahawa gris itu merupakan gris bersuhu tinggi, mempunyai penetrasi bekerja sebanyak 270-290 dan memasukkan gris itu ke dalam kategori gris umum, mempunyai kecenderungan yang rendah untuk menghakis kuprum dan konsentrasi unsur logam adalah rendah di dalam gris tersebut. Sehubungan dengan itu, boleh disimpulkan bahawa nisbah silika kesal kepada sisa pelumas lumpur dan masa campuran gris mempunyai kesan signifikan ke atas penetrasi bekerja dan titik jatuhan gris

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LIST OF ABBREVIATIONS

AAS	-	Atomic Adsorption Spectroscopy
ASTM	-	American Society For Testing and Materials
BC	-	Before Century
DSC	-	Differential Scanning Calorimetry
EP	-	Extreme Pressure
FTIR	-	Fourier Transform Infrared
KGF	-	Kilogram Force
NLGI	-	National Lubricating Grease Insitute
PSI	-	Pound Per Square Inch
RPM	-	Radium Per Minute
RUL	-	Remaining Useful Life

LIST OF SYMBOLS

^{o}C	-	Degree Celcius
F	-	Fahrenheit
ml	-	Milliliter
wt. %	-	Weight percent
%	-	Percentage

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Greases are one of the oldest forms of lubricating material and in the early days, greases are made from environmentally friendly and biodegradable materials such as mutton fat, beef fat and lime. Ancient Egyptians about 1400 B.C made crude greases to lubricate the wheels of their chariots (Rudnick, 2003). Grease is a solid to semi-solid lubricant consisting of a thickening agent dispersed in a liquid lubricant such as mineral oil (petroleum oil), synthetic oil (silicone oil) or vegetable oil. The most important factors affecting the properties and characteristics of a grease are firstly, the amount and type of thickener used, secondly, the viscosity and physical characteristics of the oil and last but not least, the additives (Bloch, 2005). Simply put, grease is made up of three essential components which are the base oil, thickener and additives.

Different types and combinations of base fluids with the thickener and additives and the way in which the grease is made will produce greases with different lubricating properties. For example, low-viscosity oil is chosen as the base oil in situations where light loads, fast speeds and low temperatures are applied whereas high viscosity base oils are for differing conditions. The thickener will determine grease properties such as water resistance, high-temperature limit, resistance to permanent structural breakdown, "stay-put" properties, and cost.

Currently, many researches were being done to search for alternative sources for grease production due to the increasing of crude oil prices in the world market and the depletion of the source. Thus, this will definitely affect the price of base oil as main component in grease. In this research, instead of using mineral oil as the base oil, silicone oil which is a more economic choice and is more abundantly available was used. To date, there is no specific research on the production of grease using waste lubricant sludge as part of its component. That is why, this research would have a significant impact in the production of grease as it proves that sludge, which is a waste could be turned into wealth and thus reducing the pollution problem and at the same time reducing the cost of the grease produced.

1.2 Problem Statement

The NLGI Grease Production Survey Report for the year 2000 reports a grand total of approximately 1.5 billion pounds of lubricating grease manufactured worldwide (Rudnick, 2003). Petroleum derived base oils currently account for about 97% of the total lubricant production (Bhaskar *et.al*., 2003). However the supply of petroleum is diminishing and thus the prices of the fuel are leaping at a significant rate. Besides being a major contributing factor to the current economy crisis , petroleum generate harmful acids which fall to earth as acid rain ,contains radioactive material and not to mention emits large amount of carbon monoxide and carbon dioxide when being combusted .In economic terms, pollution from petroleum is regarded as a negative externality. Many alternatives are being discovered to replace this limited resource. In order to overcome this problem, action should be taken to explore the utilisation of new raw materials for grease production. Therefore attempt were made in this research to study on the potential of utilising waste lubricant sludge to produce grease.

In the year 2004, 37.4 million tons of lubricants were consumed worldwide. About 50% of the lubricants sold worldwide end in and thus pollute the environment (Mang and Dresel, 2007). These wastes which were not properly disposed are toxic to the environment as it contains toxic chemicals and are not biodegradable. Waste lubricant oil contain significant amount of base oil and it is worth recovering it using solvent extraction process. However, in this study, waste lubricant sludge was recovered instead as till today; the research on the recovery of sludge is still very limited. Sludge was proven to have potential as thickener in the production of grease in this study. This step will not only reduce the disposal cost but will also produce grease at a low cost. As mentioned, using sludge as the thickener is also an incentive in this research. The reason saying so is because the thickening additive that is being used which is the fumed silica is a very good thickener but it is also very expensive. So in order to decrease the amount used in the production of grease, sludge was used to replace a significant amount of fumed silica .It is found that sludge may also have the thickening property that is needed in producing grease.

The recycling of these wastes is not only beneficial to the animals and the environment but is also important in keeping the society free from health hazards. The severity of this waste generation might not be seen now but it will definitely impact the society some time later. That is why, it is important to take the first step in recycling and turning waste to wealth no matter how small the step is. The recycling of the waste lubricating oil will stop these wastes from being improperly discarded into the environment. Besides that, this study will also be very economical when comparing to producing grease from fresh base oil. This study is also done in the hopes that society will come to realise the importance of recycling and at the same time turning waste to wealth as the treatment of wastes has become one of the most important concerns of modern society to protect the environment.

1.3 Objective

To study on the production of high temperature grease from waste lubricant sludge.

1.4 Scope of Study

In order to achieve the objective of the research study, several scopes have been identified:

1) To produce Calcium Fluoride a thermal resistant additive for grease preparation.
2) To study on the effects of using different ratios of waste lubricant sludge and fumed silica on grease characteristics. The ratios used are 100:0, 90:10, 80:20, 70:30, 60:40, 50:50 and 40:60.

3) To study on the effects of using different mixing time on grease characteristics. The mixing times used are 1, 2, 3, 4 and 5 hours.
4) To characterize the grease produced by using penetration test and dropping point test.
5) To compare the characteristics of the grease produced.

1.5 Rationale and Significance

The production of high temperature grease from silicone oil and waste lubricant sludge contributes to the community and the environment in more than one way. For starters, this research will be a stepping stone for other researches who will be doing researches similar to this and who intend to curb pollution. It will be a guide for them to what’s to do and what’s not to do in the future.

In the aspect of the society, this research will protect them from more toxic pollution of waste lubricant sludge. The protection of the environment is crucial as the cycle of life depends on it. Thus through this research, it will not only tackle the pollution problem but will also lower the chances of wildlife being exposed to these dangerous substances. It is hoped that the concept of ‘zero waste’ and ‘waste to wealth’ can be cultivated in our society for the betterment of the future.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

There are three components that form lubricating grease. These components are oil, thickener and additives. The base oil and additive package are the major components in grease formulations, and as such, exert considerable influence on the behavior of the grease. The thickener is often referred to as a sponge that holds the lubricant (Wright, 2001). Figure 2.1 shows the general composition of base oil, thickener and additive to produce grease.

There are a few reasons why greases are more favourable than oil as a lubricant. Due to its consistency, grease acts as a sealant to prevent lubricant leakage and also to prevent entrance of corrosive contaminants whereas oil would simply seep away. Besides that, grease is easier to contain than oil. Oil lubrication can require can require an expensive system of circulating equipment and complex retention devices. In comparison, grease, by virtue of its rigidity, is easily confined with simplified, less costly retention devices. Another advantage of grease is that it holds solid lubricants in suspension. Finely ground solid lubricants, such as molybdenum disulfide are mixed with grease in high temperature service (over 315^{o}C or 599^{o}F) or in extreme high-pressure applications. Grease holds solids in suspension while solids will settle out of oils. Lastly the fluid level of grease does not have to be controlled and monitored.

However, there are some disadvantages when using grease. Due to its consistency, grease cannot dissipate heat by convection like circulating oil. Grease has more resistance to motion at start-up than oil, so it is not appropriate for low torque/high speed operation. It is also more difficult to handle than oil for dispensing, draining and refilling. Also, exact amounts of grease cannot be as easily metered (Engineering Edge, 2010).

The function of grease is to remain in contact with and lubricate moving surfaces without leaking out under gravity or centrifugal action, or be squeezed out under pressure. Its major practical requirement is that it retain its properties under shear at all temperatures that it is subjected to during use. At the same time, grease must be able to flow into the bearing through grease guns and from spot to spot in the lubricated machinery as needed, but must not add significantly to the power required to operate the machine, particularly at startup (Boehringer, 1992).

Generally, a satisfactory grease for a given application must reduce friction and wear in the machine element being lubricated under various operating conditions. Besides that, a grease function as a rust and corrosion protector. Greases also prevent dirt, water and other contaminants from entering the part being lubricated and at the same time, tolerate and repel some degree of moisture so that contamination does not occur. All this happen without significant loss of performance. Many types of grease are used in an extended period of time thus it is expected to maintain its structure and consistency during long periods of use. At low temperature, a good grease permits free motion of moving parts. Furthermore, different greases have different characteristic for different method of application. A satisfactory grease should retain those characteristics during storage and not change in chemical properties or physical appearance.

Grease and oil are not interchangeable. Grease is used when it is not practical or convenient to use oil. The lubricant choice for a specific application is determined by matching the machinery design and operating conditions with desired lubricant characteristics. Grease is generally used for machinery that runs intermittently or is in storage for an extended period of time. Because grease remains in place, a lubricating film can instantly form. Machinery that is not easily accessible for frequent lubrication also requires grease. High-quality greases can lubricate isolated or relatively inaccessible components for extended periods of time without frequent replenishing. These greases are also used in sealed-for-life applications such as some electrical motors and gearboxes. Machinery operating under extreme conditions such as high temperatures and pressures, shock loads or slow speed under heavy load prefers the usage of greases compared to oils. Worn components is another suitable application for grease as grease maintains thicker films in clearances enlarged by wear and can extend the life of worn parts that were previously lubricated by oil (Engineering Edge, 2010).

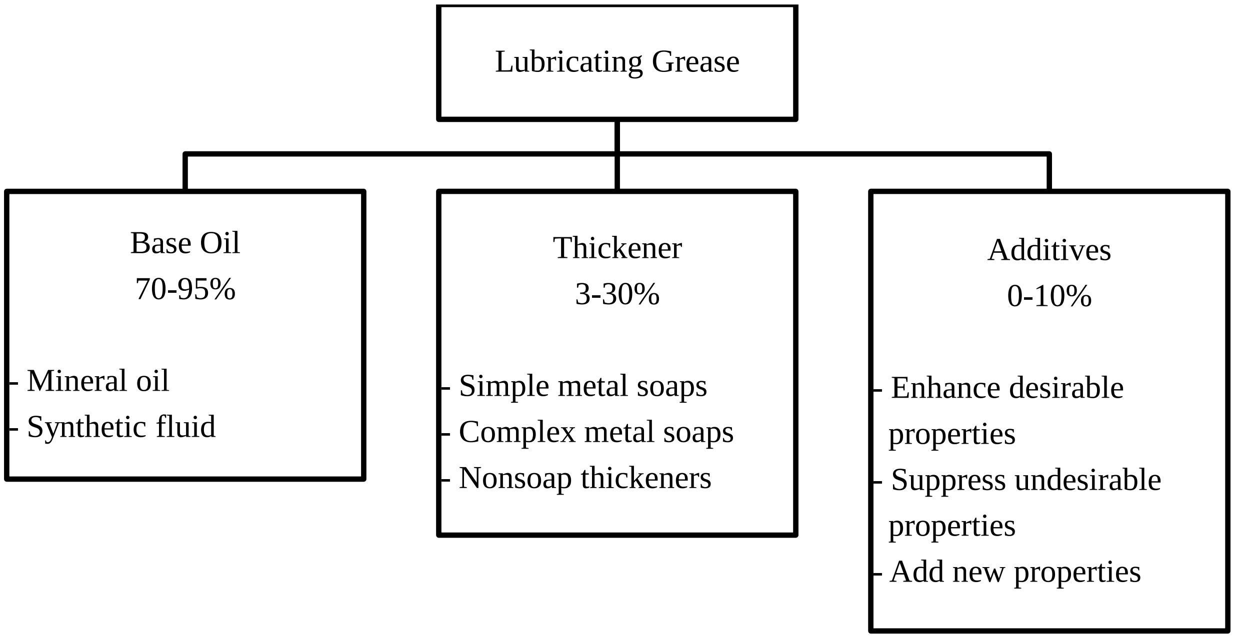

Figure 2. 1 Grease Anatomies (Wright, 2001)

2.2 Base Oils

In terms of volume, base oils are the most important components of lubricants. As a weighted average of all lubricants, they account for about +70-95% of lubricant formulations (Wright, 2003). Many different types of base oils may be used in the manufacture of grease, including mineral (petroleum), vegetable (palm oil) and synthetic oil (esters, silicones, and glycols). As mentioned before, the viscosity of the base oil is the most significant property. Lighter, lower viscosity base oil is used to formulate low temperature greases or greases suitable for high rotational speeds, while heavier, higher viscosity base oils are used to formulate greases used in applications where high loading is encountered, high temperature or low rotational speeds are seen. In this research silicone oil was used as the base oil. Silicone oil is used to produce silicone grease which is an amorphous fumed, silica-thickened, polysiloxane-based compound. Silicone greases provide lubrication, corrosion resistance and also maintain stability under high temperatures which was one of the reasons why silicone oil was chosen as the base oil in this research (Rudnick, 2006).

2.3 Thickener

Thickener is the term describing the ingredients added to the base oil in order to thicken it to a grease structure. Thickeners are, in fact what make grease grease and it is the component that differentiates grease from conventional lubricating fluid. The thickening agent may be an inorganic or an organic substance. Organic thickeners can be either soap-based or non-soap based, while inorganic thickeners are non-soap based. They are designed to reduce migration and to prevent leakage under gravity. The thickening agent makes up about 3-30% of the entire grease. Fumed silica was chosen as the thickener in this research as fumed silica has a very strong thickening effect. Waste lubricant sludge was also used as part of the thickener in this research in the effort to reduce the amount of fumed silica being used so that the cost of grease production could be lowered.

2.4 Additives

Additives are added to some grease to improve their lubricating properties. Besides enhancing performances, additive also plays a role in protecting the grease and lubricated surfaces. The most common additives found in greases are for example; antioxidants (prolong the life of a grease), anti-corrosion agent (protect metal against attack from water, sulphides or corrosive elements), Extreme Pressure agents (guard against scoring and galling) and anti-wear agents (.prevent abrasion and metal to metal contact). There are about +0-10% of additives in grease. Molybdenum disulfide and calcium fluoride (EP & high thermal resilience additives) were the additives used in this research. (Takadoum, 2008)

2.5 Types of Grease

Grease can be divided into 6 general categories or types; i.e., mixtures of mineral oils and solid materials, heavy, asphaltic-type oils blended with lighter oils, extreme-pressure greases, roll-neck greases, soap-thickened mineral oils and multi-purpose grease.

2.5.1 Mineral Oils Mixed with Solids

These types of greases are very heavy lubricants for specialized applications. Such greases lubricate rough-fitting machine parts operating under heavy pressures or loads at relatively slow speeds. Examples of equipment that will typically use this type of grease include concrete mixers, bearings and rollers on conveyors and heavy construction equipment (Hughes, 2010).

2.5.2 Heavy Asphaltic-Type Oils Blended with Lighter Oils

These types of lubricants are classified as greases but are actually thick, heavy oils used to lubricate open-type gearing and wire rope. A primary advantage of these oils is that they form a heavy protective film when heated or painted on surfaces and then allowed to cool. Lighter oil is typically blended with the heavy oils in order to improve the pour point of the oil (Hughes, 2010).

2.5.3 Extreme – Pressure Greases

The unique characteristic of this type of grease is that it contains additives to improve firm strength under various applications. In essence, film strength provides the resistance of the lubricant to being torn apart, thus preventing metal-to-metal contact of the equipment being lubricated. A film is formed by a chemical reaction of the metal to the additives in the grease. The chemical reaction is usually brought about (or accelerated) by pressure exerted on the grease, creating heat.

A few of the additives used in EP greases include compounds containing parts of chlorine, phosphorus, active and/or passive sulfur, chlorinated waxes and phosphates. Zinc and lead may also be added, as well as asbestos in some lubricants as a filler to cushion the shock loading on gear drives. The specific additive being used will always depend on the application for use. Factors to be considered for types of additives include specific equipment operating conditions such as load, speed, surface condition and inherent design characteristics (Hughes, 2010).

2.5.4 Roll Neck Greases

Roll neck greases are specialized lubricants used almost exclusively for lubricating plain bearings in rolling equipment. For example, it’s fairly common to use a block of NLGI No. 6 grease, which has the consistency of a bar of soap, carved to mate with the shape needed to accommodate the bearing of heavily loaded equipment (Hughes, 2010).

2.5.5 Soap Thicken Mineral Oils

This is by far the most widely used category of grease in industry today. This type of grease varies by the additive that forms the soap in the lubricants chemical makeup.

Sodium-base greases are also general-purpose greases. Because they have a higher dropping point (approximately 300^{o} to 350ºF), they are often used to lubricate machine parts operating near heat. Sodium greases made with lighter oils are used for ball and roller bearing lubrications, as are combinations (mixed base) of calcium and sodium grease Sodium-soap greases have a spongy or fibrous texture and are yellow or green in color. Because of their working stability and intermediate melting point, they are used for lubricating wheel bearings (other than disc brakes) and for general-purpose industrial applications. Typical examples include rough, heavy bearings operating at low speeds, as well as skids, track curves and heavy-duty conveyors.

Barium-soap greases are general-purpose types, valued for their ability to work over a wide temperature range. Their dropping point is approximately 350ºF or higher, although they are not intended to be used in continuous operation at temperatures above 275ºF. Barium-soap greases are chosen for a variety of jobs, especially for nearly all types of bearing lubrications. They have a high-soap content. However, this makes this type of lubricant less suitable for use at low temperatures and in very high-speed applications. They have a buttery of fibrous texture and are reddish-yellow or green in color.